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The use of nutshell carbons in drinking water filters for removal of chlorination by-products

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Abstract: Chlorination of drinking water is a common practice, used by numerous municipalities in the United States (US) to safeguard their water supplies. However, the chlorine used can chemically react with organic components in the drinking water to produce unwanted chlorination by-products. The objective of this investigation was to evaluate the use of granular activated carbon produced from nutshells (almond, English walnut, pecan) in a point-of-use (POU) water filtration system designated 'Envirofilter' and to determine its efficacy in removing select, potentially carcinogenic chlorination by-products, namely the trihalomethanes (THMs) bromodichloromethane, bromoform and dibromochloromethane. The POU water filtration system that contained the nutshell-based carbons was designated 'Envirofilter' and adsorption efficiencies of this system were compared to that of four commercially available POU home water filter systems, namely, BRITA, Omni Filter, PŪR and Teledyne Water Pik. Eight different 'Envirofilters' were constructed of individual or binary mixtures of carbons produced from acid-activated almond or pecan shells and steam-activated pecan or walnut shells and evaluated for adsorption of the three chlorination by-products. The results indicate that only two of the eight 'Envirofilters' failed to remove more THMs than the commercial POU systems. In both cases, these filters contained carbons with either 100% acid-activated almond shells or 100% acid-activated pecan shells. All six of the other filters contained carbons with either 50% or 100% steam-activated pecan shells or steam-activated walnut shells. Therefore, 'Envirofilters' appeared to depend on the presence of steam-activated nutshell carbons for their success. The six effective 'Envirofilters' reduced THM levels to below the Maximum Contaminant Levels (MCL) required by the US Environmental Protection Agency (US EPA). Based on these results, these six 'Envirofilters' may be considered as a replacement for existing commercial filter systems because of their efficacy and projected cost.

Keywords: granular activated carbon; point-of-use water filter; nutshells; bromoform; dibromochloromethane; bromodichloromethane

1 INTRODUCTION

Chlorine is an important water treatment chemical and is used to eliminate harmful microorganisms throughout water distribution networks, thereby preventing waterborne illnesses. However, since chlorine is a very reactive gas, chlorine addition or chlorination of drinking water yields by-products such as trihalomethanes (THMs) as the chlorine reacts with organic matter in the water. Examples of THMs include bromoform, dibromochloromethane and bromodichloromethane. THMs are known to pose potential adverse health effects, if consumed in drinking water.[1] Studies have suspected several THMs to be carcinogenic in laboratory animals and to cause adverse reproductive or developmental effects in laboratory animals.[1] They are suspected of causing health effects in humans including liver, kidney or central nervous system damage and increased risk of cancer. As such, the total THM level in drinking water was set by the US EPA at 0.080 parts-per-million or ppm (mg dm^{-3}).[1] These enforceable regulatory limits keep THMs at low levels to minimize health risks without compromising the effectiveness of chlorination or prohibitively increasing the cost of water treatment. These limits are, however, above the ideal zero THM concentration in drinking water.

Several types of commercial water treatment systems are available to the homeowner, including point-of-use (POU) and point-of-entry (POE) systems. These systems are based on one of several water purification

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methods such as activated carbon, ion exchange, reverse osmosis, and distillation. Each method has advantages and limitations. For instance, ion exchange resins are good at removing charged species such as metal ions but not the much less polar organic contaminants. The reverse is true for activated carbon. In terms of cost, reverse osmosis and distillation are costly while activated carbon is the least expensive option. Carbons, which are used in POU and POE drinking water filters, are mostly made from coal, a non-renewable resource. However, nutshell-based, granular activated carbons have been shown to exhibit excellent adsorptive properties toward a wide variety of organic molecules, including raw sugar colorants,[2–4] polar and non-polar, volatile organic compounds,[5–7] geosmin[8] and the suite of organic compounds that comprise the chemical oxygen demand (COD) of municipal wastewater.[9] All of these studies have shown that nutshell-based granular activated carbons are cost effective and are as good as, and, in some instances, even better adsorbents than the commercial carbons used for comparison. A review of the literature showed no published information on the use of nutshell-based, granular activated carbon in POU or POE home filter systems for the removal of chlorination by-products in drinking water.

The objectives of this investigation were to evaluate the adsorption efficiencies of nutshell-based granular activated carbons in home drinking water filter systems ('Envirofilters'), with respect to the adsorption of THMs, especially dibromochloromethane, bromodichloromethane and bromoform which are suspected to be carcinogenic in laboratory animals, and to compare the adsorption efficiencies of the 'Envirofilters' to the adsorption efficiencies of commercial POU home water filters.

2 MATERIALS AND METHODS

2.1 Materials

Pecan shell-based, almond shell-based and English walnut shell-based carbons were obtained from the USDA-ARS, Southern Regional Research Center, New Orleans, LA. Phosphoric acid-activated almond shells and pecan shells were produced by the method of Toles *et al*.[10] Steam-activated pecan shells and English walnut shells were developed using the procedure given by Toles *et al*.[11]

The four commercial drinking water filtration systems used in this study for comparison purposes were obtained at local retail outlets. Omni Filter (OMNI Industries, Mulvane, KS) consists of a coal-based, steam-activated carbon designed for the removal of drinking water contaminants. The active component in Teledyne Water Pik (Waterpik Technologies, Inc, Fort Collins, CO) filters is also a coal-based, steam-activated carbon used for contaminant adsorption in drinking water. PŪR water filter cartridges (Recovery Engineering, Inc, Minneapolis, MN) are designed for their pitcher filter system and contain predominantly cation exchange resin with some coal-based, granular activated carbon. They are manufactured specifically for removal of chlorine, organic compounds that contribute to bad taste and odor, sediment and various divalent metal ions. BRITA water filtration cartridges (The Brita Products Co, Oakland, CA) are similar to the PŪR water filter cartridges in both composition and intended use.

2.2 Methods

2.2.1 Determination of physical, chemical and surface properties of nutshell-based granular activated carbons

The physical properties of percent yield and bulk density and percent attrition were determined by the methods of Pendyal *et al*,[12] Ahmedna *et al*[4] and Bansode *et al*,[5] respectively. The chemical properties of pH and percent ash were measured by the procedures of Ahmedna *et al*.[4] Carbon surface properties include surface area, micropore, mesopore and macropore volume, measured by a procedure described by Toles *et al*,[13] and surface charge determined by the method of Toles *et al*.[11]

2.2.2 Preparation of 'Envirofilters' and commercial filters

The eight 'Envirofilters' were prepared according to the description provided in Table 1. In order to keep the packing dimensions of 'Envirofilters' and the four commercial filters identical to each other, product containers (filter cartridges) manufactured by BRITA were used to construct all of the filter systems. Empty BRITA containers were filled to the same filter depth and packing level for both 'Envirofilters' and commercial filters. Since the densities of the different packing materials (both experimental and commercial) were different from each other, different weights of adsorbent were present in the cartridges, as listed in Table 1. The quantity and type of adsorbent in each filter cartridge is presented in Table 1.

2.2.3 Determination of adsorption properties of the 'Envirofilters' and commercial filtration systems

Adsorption efficiency was measured by using a standard solution of trihalomethanes in ultra clean tap water. Prior to use, the tap water was filtered through a Millipore ELIX III water purification system to remove traces of organic and inorganic contaminants, if any existed. The concentrations of the standard trihalomethanes were adjusted (dibromochloromethane at $0.060\,\mathrm{mg\,dm^{-3}}$, bromodichloromethane at $0.020\,\mathrm{mg\,dm^{-3}}$ and bromoform at $0.020\,\mathrm{mg\,dm^{-3}}$) to be equal to or slightly higher than the permissible levels in drinking water allowed by the US EPA.

Prior to testing the filters, they were throughly cleaned with contaminant-free water followed by rinsing with 20% nitric acid. The ability of the filters to remove THMs was determined by following a two-step

Table 1. Filter identification and filter contents

Filter identification	Filter contents	Adsorbent in filter (g)
'Envirofilter' I	100% PA	93
'Envirofilter' II	100% PS	62
'Envirofilter' III	100% AA	73
'Envirofilter' IV	100% WS	63
'Envirofilter' V	50% PA and 50% PS	70
'Envirofilter' VI	50% PA and 50% WS	70
'Envirofilter' VII	50% AA and 50% PS	60
'Envirofilter' VIII	50% AA and 50% WS	60
Omni Filter	100% coal-based activated carbon	76
Teledyne Water Pik	100% coal-based activated carbon	74
PŪR	Mixture of cation exchange resin and coal-based activated carbon	100
BRITA	Mixture of cation exchange resin and coal-based activated carbon	96

PA, acid-activated pecan shells; PS, steam-activated pecan shells; AA, acid-activated almond shells; WS, steam-activated English walnut shells.

Table 2. Physical and chemical characteristics of nutshell-based activated carbons[a]

Agricultural by-product	Activation agent	Yield (%)	Attrition (%)	Bulk density ($g\,cm^{-3}$)	pH[b]	Ash (%)
Pecan shells	Phosphoric acid	32	45.2	0.51	3.2 (6.8)	2.1
Pecan shells	Steam	18	8.8	0.395	7.8	10.4
Almond shells	Phosphoric acid	24	60	0.42	3.3 (7.1)	7.9
English walnut shells	Steam	11	7.6	0.339	6.7	4.3

[a] Values are the means of duplicate determinations where the standard deviations are less than 10% of the mean values.
[b] Values in parentheses are adjusted pH values. The pH of the carbon was adjusted to neutrality using NaOH or HCl before its use in water filtration.

Table 3. Surface properties of nutshell-based activated carbons[a]

Agricultural by-product	Activation agent	Surface area ($m^2\,g^{-1}$)	Micropores (%)	Macropore and mesopores (%)	Surface charge (meq $H^+\,g^{-1}$)
Pecan shells	Phosphoric acid	682	92.7	7.3	2.43
Pecan shells	Steam	724	71.4	28.6	0.23
Almond shells	Phosphoric acid	708	86.6	11.4	2.46
English walnut shells	Steam	1060	81.5	18.5	0.25

[a] Values are the means of duplicate determinations where the standard deviations are less than 10% of the mean values.

pour-through, gravity-flow procedure recommended by the US EPA.[14] The pour-through filter's adsorption efficiency in removing THMs was expressed as the amount of contaminant removed per unit weight of the adsorbent with respect to unfiltered water samples in order to normalize the data.

2.2.4 Statistical analysis

The experimental design was a completely randomized design in which 12 filter types received equal volumes of the same simulated drinking water. The concentrations of THMs removed from solution were measured as dependent variables. THM adsorption was analyzed by Multivariate Analysis of Variance (MANOVA) using SAS.[15] Tukey's multiple comparison test was used to compare mean individual THM uptakes by filters within each THM category. Differences between THM adsorption of the test filters was judged significant at the $\alpha = 0.05$ significance level.

3 RESULTS AND DISCUSSION

3.1 Physical, chemical and surface properties of nutshell-based granular activated carbons

Physical, chemical and surface properties of the nutshell-based experimental carbons are presented in Tables 2 and 3. The data in Table 2 indicate that production of acid-activated pecan shell carbon resulted in the greatest percent yield compared to the yield of carbons prepared from other nutshells. Furthermore, the method of activation also had a significant effect on the percent yield regardless of the type of nutshell employed. Acid activation resulted in higher yields compared to steam activation. In regard to other characteristics, acid-activated carbons exhibited higher pH, higher percent attrition and lower bulk density than steam-activated carbons. Steam-activated carbons had higher pH and lower bulk density than acid-activated carbons. The latter had their pH adjusted to near neutrality prior to use in water filtration. The only physical or chemical property evaluated that appeared independent of activation method was percent ash, which is dependent upon the precursor used and the wash method used for ash removal after pyrolysis and activation.

These physical and chemical properties may not directly relate to a carbon's effectiveness in water purification but they are important to their commercial utilization. For example, percent yield is a factor used to estimate carbon production cost. Attrition, a measure of the mechanical strength of the carbons, may affect handling and transportation costs and regeneration. Bulk density and pH affect potential commercial use of activated carbon in terms of surface area and surface charge.

Surface area and surface charge may, however, be linked to the carbon's performance in the removal of non-polar organics such as THMs from drinking water.[3] Both surface area and the percentage of micropores versus the percentage of mesopores and macropores affect adsorption of organic molecules of different polarities and sizes. In Table 3, the highest surface area was observed in the steam-activated English walnut-based carbon. The steam-activated carbons had higher surface areas and lower microporosity, but higher meso- and macroporosity than the acid-activated carbons. The THMs used in this study are small, non-polar organic molecules that would likely depend on a non-polar surface for adsorption with a combination of micropores acting as adsorptive surfaces and meso- and macropores acting as channels to guide the THMs to the micropores.[3] Additionally, surface charge may be inhibitory to adsorption of non-polar molecules if the adsorptive surfaces contain negative charges. In Table 3, acid-activated carbons had about an order of magnitude greater negative charge than steam-activated nutshell carbons. Therefore, the likelihood of diminished adsorption due to surface charge would be greater in the acid-activated carbons than in the steam-activated carbons. Steam-activated nutshell-based carbons would appear to be more likely candidates for enhanced adsorption of small, non-polar organics than their acid-activated counterparts.

3.2 Removal of THMs by 'Envirofilters' and commercial filtration systems

The THM removal efficiencies of eight 'Envirofilters' and four commercial filters are presented in Table 4. The removal efficiencies are given as individual THM removed per unit weight (g) of adsorbent used in the filter.

Based on the data in Table 4, 'Envirofilters' II and IV–VIII were significantly better at removing all three THMs from simulated drinking water than the either the other two 'Envirofilters' (I and III) or the four different commercial filtration media on a unit weight (per g) basis. 'Envirofilters' II and IV consist of 100% steam-activated pecan shell carbon or 100% steam-activated walnut shell carbon, respectively. 'Envirofilters' I and III contained 100% acid-activated nutshell carbons and adsorbed significantly less THMs than their steam-activated counterparts. 'Envirofilters' VII and VIII, however, are binary systems comprised of 50% phosphoric acid-activated almond shell carbons and 50% steam-activated pecan or walnut shell carbons. These filters performed as well as the two single-carbon 'Envirofilter' systems. However, adsorption efficiencies with respect to THM of the 'Envirofilters' V and VI which consisted of a binary mixture of 50% acid-activated and 50% steam-activated pecan shells and 50% acid-activated pecan shells and 50% steam-activated walnut shells, respectively, were less than the 'Envirofilters' VII and VIII which also contained binary mixtures differing in the precursor used and methods of activation of the components in the binary mixture (Tables 1 and 4).

Since 'Envirofilters' II and IV consisted of exclusively steam-activated carbons (Table 1), a reason for

Table 4. Amount of chlorination by-products removed per unit mass of adsorbent[a]

Filter identification[b]	Adsorbent in cartridge (g)	Dibromochloromethane (μg/l/g)	Bromodichloromethane (μg/l/g)	Bromoform (μg/l/g)
'Envirofilter' I	93	0.561[e]	0.215[e]	0.207[f]
'Envirofilter' II	**62**	**0.963**[a]	**0.322**[a]	**0.310**[b]
'Envirofilter' III	73	0.797[c]	0.263[d]	0.267[d]
'Envirofilter' IV	**63**	**0.946**[a]	**0.317**[a]	**0.314**[b]
'Envirofilter' V	**70**	**0.852**[b]	**0.286**[b]	**0.272**[c]
'Envirofilter' VI	**70**	**0.851**[b]	**0.286**[b]	**0.272**[c]
'Envirofilter' VII	**60**	**0.968**[a]	**0.319**[a]	**0.323**[a]
'Envirofilter' VIII	**60**	**0.963**[a]	**0.320**[a]	**0.326**[a]
Omni Filter	76	0.683[d]	0.263[d]	0.252[e]
Teledyne Water Pik	74	0.805[c]	0.270[c]	0.267[d]
PŪR	100	0.596[e]	0.200[g]	0.199[g]
BRITA	96	0.551[e]	0.208[f]	0.200[g]

[a] Values are the means of duplicate determinations where the standard deviations are less than 5% of the mean values. Values with different superscripts within each column are significantly different across different filters used. Values in bold represent THM concentrations removed that are greater than THM concentrations removed using any of the four commercial water filters. Contaminant concentrations used were dibromochloromethane (60 μg dm^{-3}), bromodichloromethane (20 μg dm^{-3}), and bromoform (20 μg dm^{-3}).
[b] See Table 1 for filter contents.

their excellent performance could be based on the explanation given in the previous section. That is, a high surface area coupled with low surface charge and well-developed meso- and macroporosity exhibited by both steam-activated pecan and walnut shell carbons could encourage adsorption of small, non-polar organic molecules, such as the THMs used in this study. The presence of high surface area and well-developed meso- and macroporosity would allow faster diffusion of liquid into the pore structure of the carbon and a greater opportunity for physical adsorption of THMs to occur within the micropores. However, in the binary system that exists in 'Envirofilters' VII and VIII, excellent THM adsorption was also achieved (Table 4). These results are harder to reconcile, but may be due to the lack of THM saturation of the steam-activated carbons in 'Envirofilters' II and IV. Although their amounts are reduced by 50% in 'Envirofilters' VII and VIII, they still have sufficient adsorption sites to achieve the high adsorption observed for these filter systems in Table 4.

Regardless of the reason, binary mixtures of carbons consisting of acid-activated almond shells with either steam-activated pecan or walnut shells removed significant quantities of THMs. The excellent THM uptake exhibited by these binary carbon mixtures is highly significant since their acid-activated portion has high surface charge and could, therefore, be favorable for adsorption of metal ions, in addition to chlorination by-products, from water.[4] This may enable an 'Envirofilter' to remove organic and inorganic contaminants with 100% agricultural by-product-based activated carbons. A properly constructed 'Envirofilter' mimics the PŪR and BRITA filtration systems in that steam-activated nutshell carbons will assume the role of steam-activated, coal-based carbons while acid-activated nutshell carbons will assume the role of cation exchange resins in these commercially available water filters made from mixtures of cation exchange resin and coal-based activated carbon.

3.3 Estimated product cost for 'Envirofilter' and commercial POU water filtration systems used in this study

Table 5 estimates the product cost for the two types of 'Envirofilters' that were the top performing filtration systems in this study (Table 4). The estimated cost consists of raw material costs (nutshells), carbon production costs, which are based on estimated costs from Toles et al[16,17] for both acid-activated and steam-activated nutshell carbons, plastic filter manufacturing costs based on an estimate given by a plastics manufacturer and miscellaneous costs, that include advertising, and transportation costs. For the single carbon systems ('Envirofilters' II and IV), we estimate the total cost to be \$8.29 per kg of carbon and for the binary carbon systems ('Envirofilters' V–VIII), the cost is slightly higher at \$8.72 per kg of carbon.

Table 5. Estimated production cost to manufacture 'Envirofilters' II, IV or VII, VIII

Item	Cost (\$ per kg of carbon) 'Envirofilters' II or IV	'Envirofilters' VII or VIII
Nutshells	0.15[a]	0.12[a]
Carbon production	1.54[b]	2.00[b]
Plastic cartridge production	6.30[c]	6.30[c]
Miscellaneous costs	0.30[d]	0.30[d]
Total production cost	8.29	8.72

[a] Nutshell cost is estimated to be \$0.09 kg^{-1} carbon for almond shells based on a 24% yield and \$0.15 kg^{-1} carbon for pecan or walnut shells based on a 15% yield.
[b] Carbon production cost is based on the cost to steam activate nutshells (\$1.54 kg^{-1} carbon) or acid activate almond shells (\$2.45 kg^{-1} carbon) which is derived from information provided by Toles et al.[16,17]
[c] Cost based on information supplied by a manufacturer of plastic cartridges and assuming there are nine pitcher filters per kg of carbon, since each pitcher filter contains 0.11 kg of adsorbent. One filter cartridge was estimated to cost \$0.70.
[d] These costs include advertising and shipping costs to retail outlets.

Table 6. Production and retail cost comparisons between 'Envirofilter' VII or VIII and the two leading commercial water filtration systems

Filtration system	Cost (\$) per kg of adsorbent	Cost (\$) per POU filter
'Envirofilters' II or IV	8.29[a]	0.92[b]
'Envirofilters' VII or VIII	8.72[a]	0.97[b]
PŪR	77.94[b]	8.66[c]
BRITA	71.91[b]	7.99[c]

[a] Based on cost data provided in Table 5.
[b] Based on 0.11 kg of adsorbent per POU filter. Therefore, one kg of adsorbent would fill nine filter cartridges.
[c] Retail cost of each pitcher filter.

Cost comparisons between the two types of 'Envirofilter' and two commercial POU water filtration systems are given in Table 6. BRITA and PŪR were selected because they are the two most popular brands of drinking water filtration systems in the United States. Two costs are generated. First, is the estimated cost per kg of adsorbent, where a production cost is listed for 'Envirofilter' and retail costs are given for the commercial products. Second, is the estimated cost per pitcher type filter unit. In both cases, the cost for the two types of 'Envirofilter' is considerably less than the retail prices for the commercial products. This cost differential would allow an 'Envirofilter' manufacturer to adjust the wholesale and perhaps the retail price of the product over a wide range. We give no estimate of a retail cost for the 'Envirofilters' because every manufacturer would have a specific target 'return on investment' figure they would use to price the filtration unit. However, a manufacturer could sell 'Envirofilters' at a retail cost similar to the commercial units, since they appear to be more efficient at removal of chlorination by-products on a per unit weight basis than the potential competition.

4 CONCLUSIONS

The results of this investigation have shown that the THM adsorption efficiency of six out of eight 'Envirofilters' described in this study were greater than that for the commercial brand filters. Furthermore, estimated manufacturing costs show that 'Envirofilters' can be manufactured at a lower cost compared to retail costs of commercial filtration units. The preceding observations lead to the conclusion that 'Envirofilters' have the potential to compete with commercial filters or at a minimum, serve as alternates to current commercial filters used for removing chlorination by-products in drinking water. The result of this study also show that, among the various 'Envirofilters' tested, that filters with steam-activated nut shell-based carbons possessed the most desired surface area and porosity (factors which are responsible for maximum removal of chlorination by-products in drinking water) and the lowest manufacturing cost, making them the best choice among the experimental filters. Additional advantages of the 'Envirofilters' include that the feedstock used to make the carbons (nutshells) are 'home grown' plant-based materials, always available in abundance, renewable and not subjected to unpredictable international economic and market conditions that affect imported feedstock such as coconut shells.

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